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A NEW METHOD FOR THE ANALYSIS OF VOLATILE CORROSION INHIBITED PAPER



TECHNICAL REPORT

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ABSTRACT

The Government procures and stores large quantities of volatile corrosion inhibited (VCI) paper until it is needed to package and preserve materiel. Since the paper's usefulness decreases with age a rapid test method was developed which would indicate the present condition of the stored paper.

The method utilized a commercial instrument, the Corrater, which measures instantaneous corrosion rates in a conducting media. Initially, measurements were made to determine what effect additions of VCI to a salt solution would have on the corrosion rate of mild steel. A correlation was established between the decrease in the corrosion rate and VCI concentration. Standardization curves were obtained for dicyclohexylammonium mitrite and sodium nitrite, which are currently used in VCI paper.

Laboratory and field tests were conducted on new and used VCI paper to establish the reliability and accuracy of the method. The VCI papers were analyzed by adding a paper sample to the salt solution, stirring to remove the VCI, and noting the decrease in the corrosion rate. The decrease in the corrosion rate was converted to weight of loading (grams per square foot) by means of the standard curves previously prepared. The results obtained compared favorably with results obtained by other more tedious analytical methods.

The method developed has the following advantages:
(1) utilizes a stable reagent, (2) nontechnical personnel can conduct the test, (3) the instrument is battery operated and (4) the test time is less than one hour.

FOREWORD

The work reported here was performed under DA Project No. 1CO24401A109, AMS Code 5025.11.803, "Corrosion Preventives and Specialty Compounds." It was carried out for the purpose of developing more reliable accelerated laboratory tests for the evaluation of corrosion preventives.

The author wishes to thank G. A. Marsh of Pure Oil Research Center for his helpful suggestions during this work.

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PROBLEM

To develop a field test for determining the usefulness of stored VCI paper.

BACKGROUND

VCI paper has been in use by the various Government agencies for a number of years to package and preserve materiel. Procurement in large quantities has required that the sheets and rolls be stored until needed. Since the paper's usefulness depends on the volatile component, it is a matter of concern whether VCI paper would still be effective if stored for six months or longer. Therefore, a rapid test method is needed which will indicate the present condition of the paper.

There are two general methods which have been used to evaluate the usefulness of the VCI paper. (a) A chemical analysis of the volatile component in the paper, and (b) the use of the corrosion of a metal specimen as an indication of the condition of the paper.

Investigators, using the chemical analysis method, would calculate the weight of loading of the paper. Using this data together with corrosion observations they could determine the weight of loading value below which corrosion would occur. Wachter, for example, assumed the protective life of packages involving 2 gram loaded dicyclohexylammonium nitrite paper was compromised when the weight of loading declined to a value of 0.1 grams per square foot. The weight of loading was determined by chemical analysis for nitrite content.

Assuming that the minimum weight of loading could be determined by corrosion tests, the chemical analysis of loading is difficult to determine since the VCI papers contain various inhibitors. These methods are reviewed below for some of the more common inhibitors.

Paper A (dicyclohexylammonium nitrite)

The total nitrite content can be determined with the method suggested by Johnson and LeMar. This analysis will indicate the volatile and non-volatile nitrites and may not be accurate since there might be other nitrites present in the paper. LeMar has developed a rapid turbidimetric procedure for analyzing the dicyclohexylamine content.

Paper B (Sodium nitrite, urea, diethyl-ethanolamine benzoate)

The total nitrite can be determined with the method suggested by Johnson and LeMar. (2) The urea content can be determined turbidimetrically after the formation of a urea-diacetyl complex in a method developed by LeMar. (4) Benzoate content can be estimated by Roberts method. (5)

Paper C (Dicyclohexylammonium caprylate, morpholine caprylate)

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The caprylic acid content can be determined by titrating an isopropyl alcohol extract with sodium hydroxide. (6)

All of the above methods are accurate, however, they have distinct disadvantages. These include (a) the necessity of special reagents and chemicals which deteriorate on storage, (b) they require trained laboratory personnel to carry out tests, and (c) no single method is applicable to all of the VCI papers.

The above considerations led Roller⁽⁷⁾ to the study of using the corrosion of a metal specimen to indicate the depletion of the paper.

This method involves the use of visual and electrical corrosion indicators of thin metal films which are sensitized to corrode at a rate slightly greater than that of the metal being protected. Results to date, (8) utilizing them as indicators for VCI packages, are not reproducible and require further study.

Since none of the previously mentioned methods offered an answer to the problem, another approach was investigated. This involved the use of an instrument called the Corrater. Work conducted by Marsh (9) has indicated the Corrater to be a useful tool for determining the corrosion rates of metals in an aqueous corrosive media.

The apparatus is now in commercial production under the trade name Magna Corrater. The instrument has two identical electrodes made of the metal to be studied. When a voltage is applied across the electrodes, which are immersed in a conductive liquid, a current will flow from one electrode. In ough the liquid to the other electrode. This current is measured in one direction, then by changing the polarity of the electrodes the current is measured in the other direction. The average of the two current readings can be converted to the corrosion rate (mils/yr.) by means of a conversion chart.

It has also been indicated in other work by Marsh (10) that the corrosion rate of a metal in a salt solution is There is then dependent on the amount of inhibitor added. a relationship between the decrease in the corrosion rate and the concentration of the inhibitor. The relationship or curve can be determined by adding known amounts of an inhibitor to a corrosive media and the decrease in the corrosion rate noted. As an example, an unknown amount of inhibitor present in a piece of VCI paper when added to the corrosive media would cause a certain corrosion rate change. This decrease in corrosion rate would correspond to a known amount of inhibitor on the curve previously plotted. In this way, the Corrater could be used to determine quantitatively, the amount of inhibitor present in any VCI paper.

APPROACH

For this investigation, the Corrater was utilized to determine the effect of additions of VCI on the corrosion current of mild steel in an aqueous corrodent. The decrease in the corrosion current was related to the VCI concentration. In this manner, standard curves were prepared relating the decrease in the corrosion current to VCI concentration. New and used VCI paper was analyzed using the Corrater and an established analytical method. This established the reliability and accuracy of the method.

The apparatus used for this study is shown in Figure 1. It consists of the Corrater and the metal probe assembly immersed in the salt solution. The solution was agitated by means of the magnetic stirrer.

The materials used for this study are listed below:

- 1. 0.01% NaCl Solution.
- 2. Electrode assembly consisting of 2, 1/4" by 3" SAE 1020 mild steel electrodes separated 1/4" by a rubber stopper.
- 3. Pure Oil Corrosion Rater (Type 3).
- 4. VCI Paper Samples
 - A Dicyclohexylammonium nitrite
 - B Urea, sodium nitrite, diethylethanolamine benzoate
 - C Dicyclohexylamine caprylate, morpholine caprylate

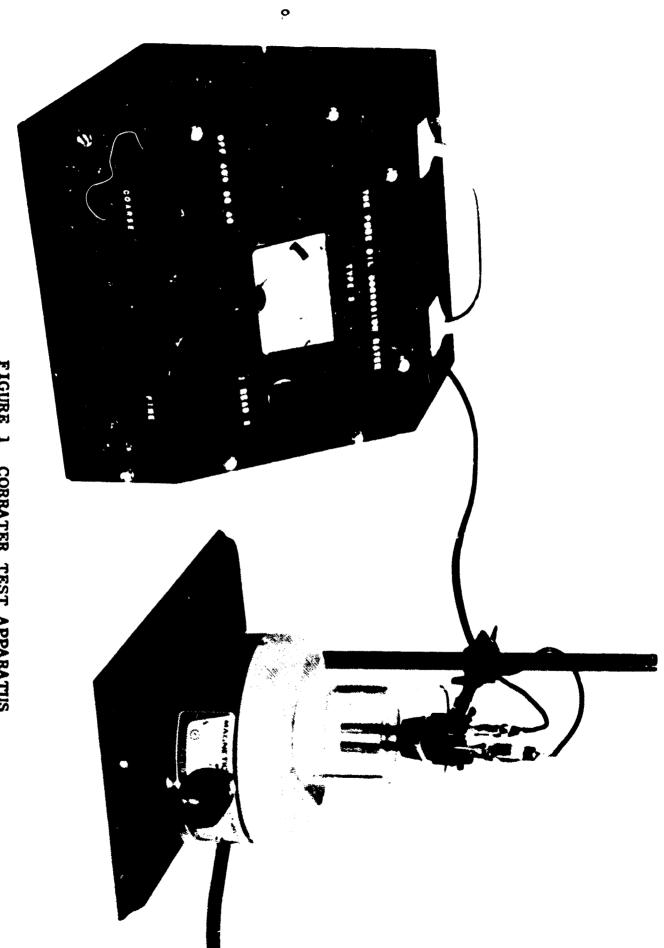


FIGURE 1 CORRATER TEST APPARATUS

5. VCI Chemicals

- A Dicyclonexylammonium nitrite
- B Urea
- C Sodium nitrite
- D Diethylethanolamine benzoate

The procedure used in making test measurements was as follows:

- l. The electrodes were prepared for test by sand-blasting and immediately immersing in methanol (conforming to O-M-232). The methanol was heated above the dew point and the electrodes were removed. Further cleaning was accomplished by spraying with naphtha (conforming to TT-N-95) followed by a rinse in hot naphtha and hot methanol. After the electrodes were dry they were placed in a dessicator until used.
- 2. 100 ml of 0.91% Sodium Chloride (NaC1) solution was added to a 150 ml beaker and placed on a magnetic stirrer.
- 3. The electrodes were placed in the salt solution and the magnetic stirrer and timer started at the same time.
- 4. The leads from the Corrater were connected to the electrodes and the readings were taken as follows:
 - a. The range switch was turned on to the 40 microampere range.
 - b. The selector switch was turned to position l and the coarse and fine adjustments were made until the meter read zero.
 - c. The selector switch was changed to read and the corresponding reading on the meter was noted.
 - d. Steps b and c were repeated for the selector switch position 2.

5. Calculation

a. The average reading was obtained by summing the 1 and 2 readings and dividing by two.

b. Example: 1 read = $\frac{28}{2}$ read = $\frac{32}{60}$

average reading = $\frac{60}{2}$ = 30

- 6. Average readings were made every 5 minutes up to 15 minutes.
- 7. At a time of 15 minutes, a known amount of inhibitor or one square inch of VCI paper was added to the salt solution.
- 8. Average readings were taken every 10 minutes up to 70 minutes.

RESULTS AND DISCUSSION

For this work, three VCI papers were investigated representing three basic formulations on the Qualified Products List for MIL-P-3420. The results of adding one square inch samples of each of the VCI papers to a .01% salt solution are shown in Figure 2. Papers A and B caused a decrease in the corrosion current and came to steady current after about 70 minutes. Paper C, however, did not decrease the current when added at 15 minutes, so the sample was added at a time of 1 minute. The results show some decrease in the current but the current begins to rise after 45 minutes. Unsatisfactory results are probably due to the incomplete removal of the compound from the paper.

To obtain a calibration curve, a solution of each inhibitor was prepared and known amounts of the inhibitor were added to a 0.01% NaCl solution. The decrease in corrosion current was measured for different amounts of the inhibitor. The decrease in corrosion current values were plotted against the concentration values to obtain a standard curve.

In the next section the details for the investigation of each VCI paper are given.

Paper A

This VCI paper, when manufactured, contained approximately 2 gms/sq.ft. of dicyclohexylammonium nitrite. For this work, 1 sq.in. of paper was used. Therefore, it will contain 0.014 grams of the inhibitor. To prepare the calibration curve, amounts of the inhibitor ranging from 0.7 mg. to 14 mg. were added to a 0.01% NaCl solution. The

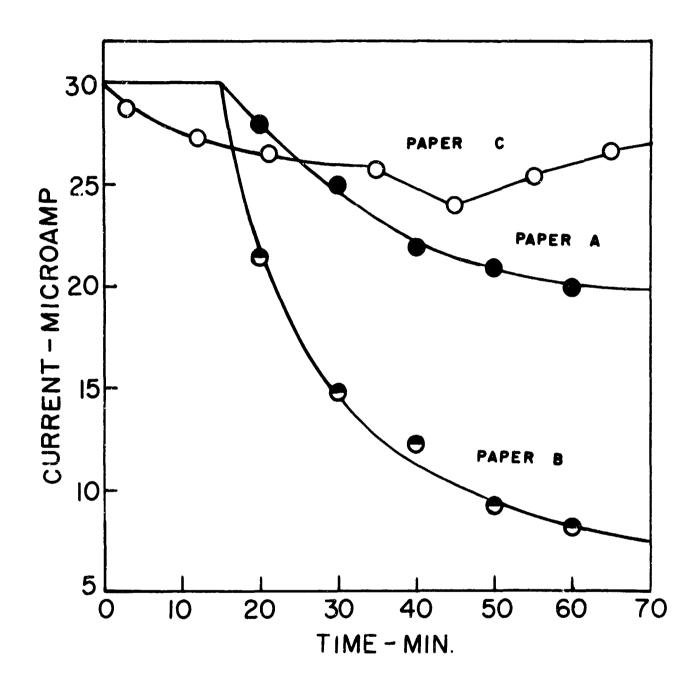


FIGURE 2 EFFECT OF VCI'S ON THE CORROSION CURRENT

curves in Figure 3 indicate a definite relationship between the amount of inhibitor added and the maximum decrease in the current. This relationship is shown in Figure 4 where the maximum current decrease is plotted against the amount of inhibitor added. Inhibitor amounts in ppm can be converted into weight of loading values in grams per square foot by multiplying ppm times 0.0144.

To test the reliability and reproducibility of this method, new and used samples of Paper A were obtained and analyzed. New samples of Paper A were conditioned so they would be at various stages of depletion. This was accomplished by placing new paper in a 130° oven for various lengths of time. The paper samples were analyzed by the Colorimetric (2) and Corrater methods. Table I indicates the weight of loading values obtained for the paper samples. Results show that the Corrater is an effective tool for analyzing paper A.

A field test was also conducted on VCI paper A being used and stored in the Storage Branch of the Rock Island Depot Activity. The results of this test are indicated in Table II. The VCI papers tested ranged in weight of loading from 1.02 to 1.89 grams per square foot. Samples were also tested in the laboratory using the Colorimetric method. Results compared favorably with values obtained utilizing the Colorimetric method.

Paper B

This VCI paper was composed of a mixture containing urea, sodium nitrite, and diethyethanolamine benzoate. A freshly manufactured paper would contain 0.05, 1.0, and 0.95 grams, respectively, to give a total weight of loading of 2 grams per square foot.

The results obtained in Figure 2 indicated that this paper caused the greatest decrease in the corrosion current as compared to the other papers. This effect was probably due to the sodium nitrite as it is a powerful inhibitor when used in an aqueous media. To investigate this assumption, amounts of urea, sodium nitrite and diethylethanolamine benzoate equal to that in one square inch of a 2 gram loaded paper were added separately to 0.01% NaCl solutions and the effects of each were noted. Urea and diethylethanolamine benzoate had no effect on the current after 70 minutes but the sodium nitrite caused a decrease in current equal to that of the new paper. Therefore, the condition of Paper B was based on the amount of sodium nitrite in the paper.

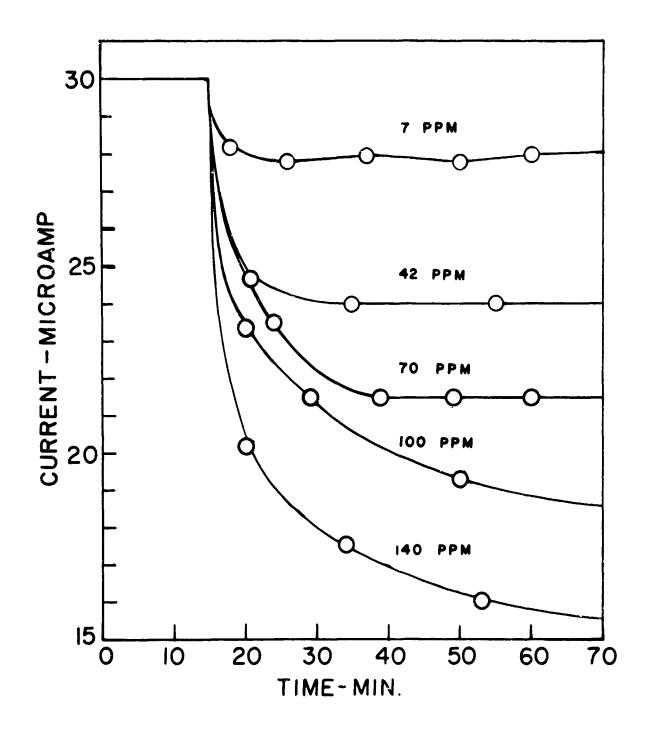
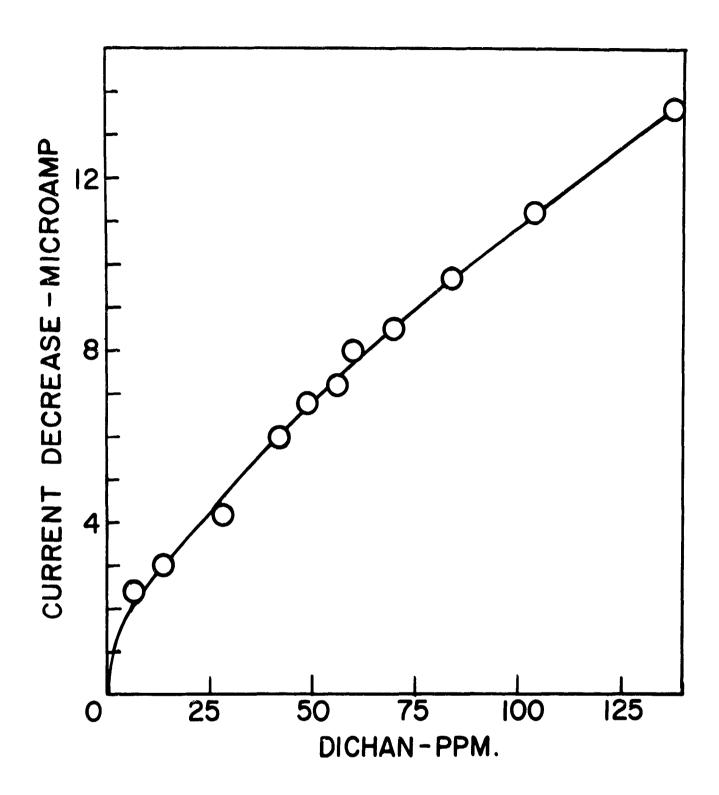


FIGURE 3 EFFECT OF DICYCLOHEXYLAMMONIUM NITRITE ON THE CORROSION CURRENT



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STANDARD CURVE FOR DICYCLOHEXYLAMMONIUM NITRITE

FIGURE 4

TABLE I
RESULTS OF THE ANALYSIS OF OVEN TREATED VCI PAPER A

Time in	Wt. of loading (gms/sq.ft.)			
Oven (hrs.)	Corrater	Colorimeter		
0	1.26	1.45		
6	0.90	0.90		
16	0.61	0.35		
42	0.09	0.09		

TABLE II

RESULTS OF ANALYSIS OF VCI PAPER A OBTAINED FROM ROCK ISLAND DEPOT ACTIVITY

Sample	Date Mfgd.	Wt. of Loadin Corrater	g (gms/sq.ft.) Colorimeter
Bag Liner	3-64	1.87	1.90
Packaged Item	3-59	1.15	1.05
Cut Pieces	1-64	1.45	1.39
Roll Being Used	1-64	1.55	1.23
Packaged Item	7-63	1.02	0.89
Packaged Item	5-64	1.70	1.90
Roll In Storage	12-63	1.89	1.76
Roll In Storage	-	1.64	1.62

In order to prepare the calibration curve various amounts of sodium nitrite ranging from 0.7 mg. to 7 mg. were added to a 0.01% NaCl solution. The time versus the corrosion current data is shown in Figure 5. The maximum decrease in the corrosion current was obtained from these curves and plotted in Figure 6 against the concentration of the inhibitor.

Results shown in Figure 2 indicate that 1 sq. in. of new paper B containing 7 mg. of sodium nitrite caused a decrease in the corrosion current of 22. Similar results can be obtained when 7 mg. of sodium nitrite is added as shown in Figure 6. This indicates that the method can be used to determine the weight of loading of new Paper B.

In the case of depleted paper, Sherk and Roberts (11) have shown that the two components, ethanolamine benzoate (used in an earlier formulation) and sodium nitrite, volatilize in equimolar quantities. It was also indicated that the paper failed to pass the VM test (12) when the weight of loading dropped to 0.2 gm/sq.ft. This point of failure coincided with the depletion of the ethanolamine benzoate from the paper leaving the sodium nitrite and urea behind.

Assuming that we have the same situation in this case, we can calculate the expected point of failure. For example, if a fresh sample of Paper B has a weight of loading of 2 gms/sq.ft. containing approximately 0.05 gms of urea, 1 gm of sodium nitrite and 0.95 gms of diethylethanolamine benzoate. Then calculating the number of moles of each we have,

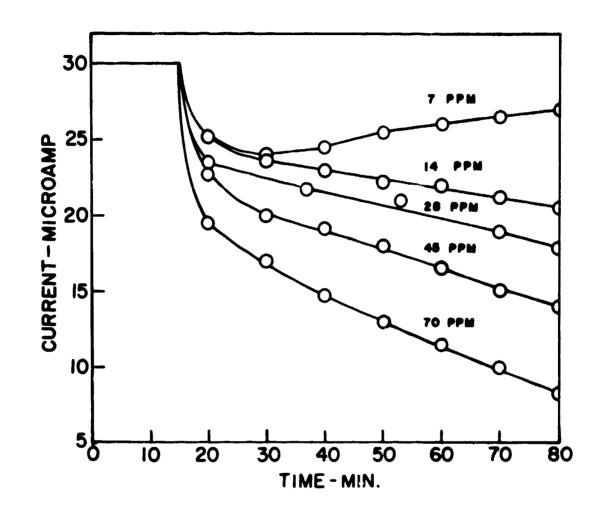
	mol. wt.	gms.	moles
urea	60	0.05	0.0008
sodium nitrite	69	1.0	0.0144
diethylethanolamine benzoate	239	0.95	0.0039

If we have a mole for mole loss of sodium nitrite and diethylethanolamine benzoate, then,

moles sodium mitrite = .0144

moles diethylethanolamine benzoate = .0039 .0105

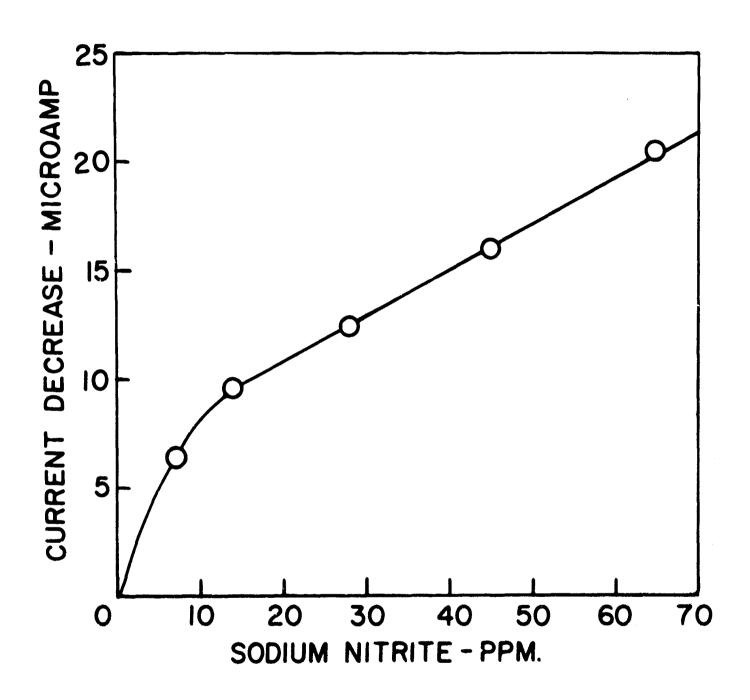
.0105 X 69 = 0.72 gms sodium nitrite left on paper



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EFFECT OF SODIUM NITRITE ON THE CORROSION CURRENT

FIGURE 5



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FIGURE 6 STANDARD CURVE FOR SODIUM NITRITE

This would indicate that Paper B should fail to protect metals from corrosion when the weight of loading of the sodium nitrite drops to 0.72 gms/sq.ft.

The following tests were made to confirm this theoretical end point. Samples of Paper B were placed in a 130°F oven and removed at various stages of depletion. The paper samples were analyzed for the sodium nitrite content with the Corrater and the Colorimeter. The results are shown in Table III. The weight of loading values by the Corrater agree well with the results obtained colorimetrically. To test the ability of the papers to still prevent corrosion of steel the Vapor Inhibitor Ability Test was performed according to MIL-P-3420B. In Table III the results of the VIA test indicated that the paper sample with an average weight of loading of 0.74 gms/sq.ft. failed the test. These results show that the Corrater can be used to effectively indicate the point of failure of Paper B.

TABLE III

RESULTS OF THE ANALYSIS OF OVEN TREATED VCI PAPER B

Time in	Wt. of (gms/		
Oven (hrs.)	Corrater	VIA Test	
0	1.04	1.09	Passed
168	0.82	0.89	Passed
264	0.70	0.78	Failed
300	0.63	0.70	Failed

A field test was not performed on this paper because of the lack of its use at this installation.

Paper C

This VCI paper contains two components in equal quantities, dicyclohexylamine caprylate and morpholine caprylate to give a total weight of loading of 2 grams per square foot.

The results shown in Figure 2 indicated some of the problems encountered with this paper. The main

difficulty was the removal of the two components from the paper. A waxy substance used to apply the compounds prevented the complete removal using the salt solution.

Further study is needed on this paper in order to resolve some of these problems.

CONCLUSIONS AND RECOMMENDATIONS

Results of this investigation have shown that the Corrater is an effective tool for the evaluation of VCI papers of the types A and B. The method of analysis is fairly simple, and rapid and is easily adapted for field use. Therefore, it is recommended that this new method of analysis be used as a field inspection procedure for the analysis of stored VCI Papers A and B.

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